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Allerston, Laura K.; Rees, Neil V.

DOI:

[10.1016/j.coelec.2018.03.020](https://doi.org/10.1016/j.coelec.2018.03.020)

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*Document Version*

Publisher's PDF, also known as Version of record

*Citation for published version (Harvard):*

Allerston, LK & Rees, NV 2018, 'Nanoparticle impacts in innovative electrochemistry', *Current Opinion in Electrochemistry*, vol. 10, pp. 31-36. <https://doi.org/10.1016/j.coelec.2018.03.020>

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## Review Article

# Nanoparticle impacts in innovative electrochemistry

Laura K. Allerston<sup>1</sup> and Neil V. Rees<sup>2,\*</sup>



Recent developments in the use of nanoimpacts as an investigative electrochemical technique are discussed. Highlights include literature on the imaging of nanoimpacts themselves and developments in the application of nanoimpacts such as, surface analysis of single particles including surface coverage and sizing. Also included are factors to consider which may affect the outcome of nanoimpact experiments.

### Addresses

<sup>1</sup> Centre for Doctoral Training in Fuel Cells and their Fuels, School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, United Kingdom

<sup>2</sup> School of Chemical Engineering, University of Birmingham, Edgbaston, Birmingham B15 2TT, United Kingdom

\*Corresponding author: Rees, Neil V. ([n.rees@bham.ac.uk](mailto:n.rees@bham.ac.uk))

Current Opinion in Electrochemistry 2018, 10:31–36

This review comes from a themed issue on **Innovative Methods in Electrochemistry**

Edited by **Frank Marken**

For a complete overview see the [Issue](#) and the [Editorial](#)

Available online 26 March 2018

<https://doi.org/10.1016/j.coelec.2018.03.020>

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## Introduction

The nanoimpact method provides analysis of single particles by detecting transient current peaks which are produced when solution-borne nanoparticles come into contact with an electrode which is held at a suitable electrical potential to either: (i) cause direct oxidation/reduction of the nanoparticle, or (ii) cause the oxidation/reduction of an electroactive solution species at the surface of the nanoparticle. The initial observation of the nanoimpact phenomenon was made by Micka in the 1950s [1], and revived by Heyrovsky *et al.* in 1995 [2–5], and again by Bard and Xiao in 2007 [6]. Following this, extensive research has been conducted in the rapidly expanding field. This review will discuss recent advances in the technique since 2015, and the interested reader is directed to several reviews covering the pre-2015 period [7–9].

## Imaging

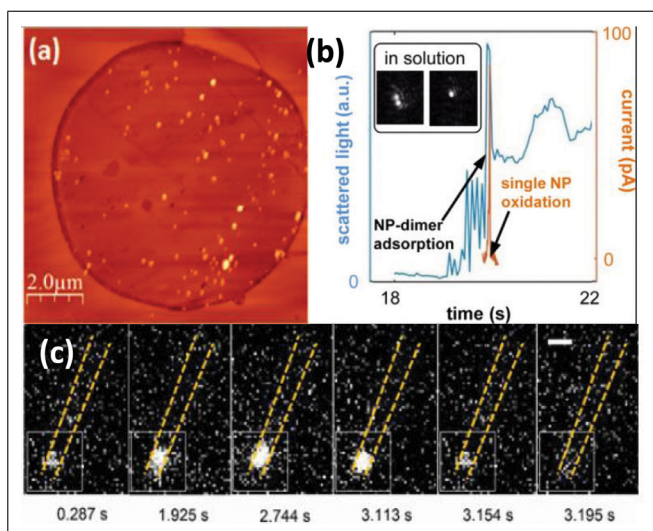
Electrochemical interfaces are challenging to study at the nanoscale although imaging of these surfaces can provide significant insight into their nature. AFM has recently been used to confirm the number of Pt nanoparticles (NPs) observed experimentally on an Au microelectrode and the number of collisions that resulted in their adsorption [10<sup>•</sup>]. These NPs were then investigated using hydrazine oxidation via electrocatalytic amplification: the AFM results combined with TEM and dynamic light scattering (DLS) showed that each individual adsorbed particle on the ultramicroelectrode (UME) was electrochemically active [10<sup>•</sup>]. The success of collisions should be viewed in the light of evidence that Ag NPs have been used to show that particles can collide more than once, causing multiple peaks in a chronoamperogram [11–13<sup>••</sup>]. Fluorescence imaging has also been utilised to capture impacts using a nanocell, where a Pt NP was deposited at the tip of a quartz bipolar nanoelectrode [14<sup>•</sup>]. The nanocell provides a small area for Ag NPs to collide with the Pt NPs and be oxidised, allowing the process to be analysed in a 1D space with single particle fluorescence microscopy [14<sup>•</sup>]. Optical imaging has also been used to monitor agglomeration of Ag NPs in solution [15]. Brasiliense *et al.* [15] combined holographic microscopy with impacts to investigate NP agglomeration and their resulting current impact response. Figure 1b below shows how optical imaging allows the agglomeration of the nanoparticles to be seen which cannot easily be detected electrochemically. When the agglomerate reached the UME it was only partially oxidised (confirmed both by the charge and optical images) which suggests that agglomerates may not behave electrochemically as larger particles [15].

## Soft particles (enzymes and DNA)

Biosensing could also be an important application for nanoimpacts; having already been used as a technique for DNA analysis [16,17]. Early nanoimpact work reported that DNA immobilised on Pt NPs detected far fewer impacts than expected [17], but by adding an enzyme to the solution, a small part of the DNA is removed, resulting in more charge peaks, of around half of the expected maximum peak current (see Figure 2 [18<sup>•</sup>]).

Chan *et al.* [19] used impacts to characterise novel individual silicon nanoparticle–enzyme hybrids which can be used in areas such as the synthesis of new biocatalysts and drugs. Their experiment used a chronoamperometric measurement at a potential where oxygen is produced by a hybrid and compared this to unmodified SiNPs. When

Figure 1



(a) AFM image of UME after NP impacts [10<sup>•</sup>]. Reprinted with permission from Ref. [10<sup>•</sup>]. Copyright (2017) American Chemical Society. (b) Correlated EC and optical signals for Ag NP agglomeration formation followed by oxidation when adsorbed onto the UME [15]. Reprinted with permission from Ref. [15]. Copyright (2016) American Chemical Society. (c) Fluorescence images of a single NP at different times interacting with Pt electrode in the nanocell [14<sup>•</sup>]. Reprinted with permission from Ref. [14<sup>•</sup>]. Copyright (2017) American Chemical Society.

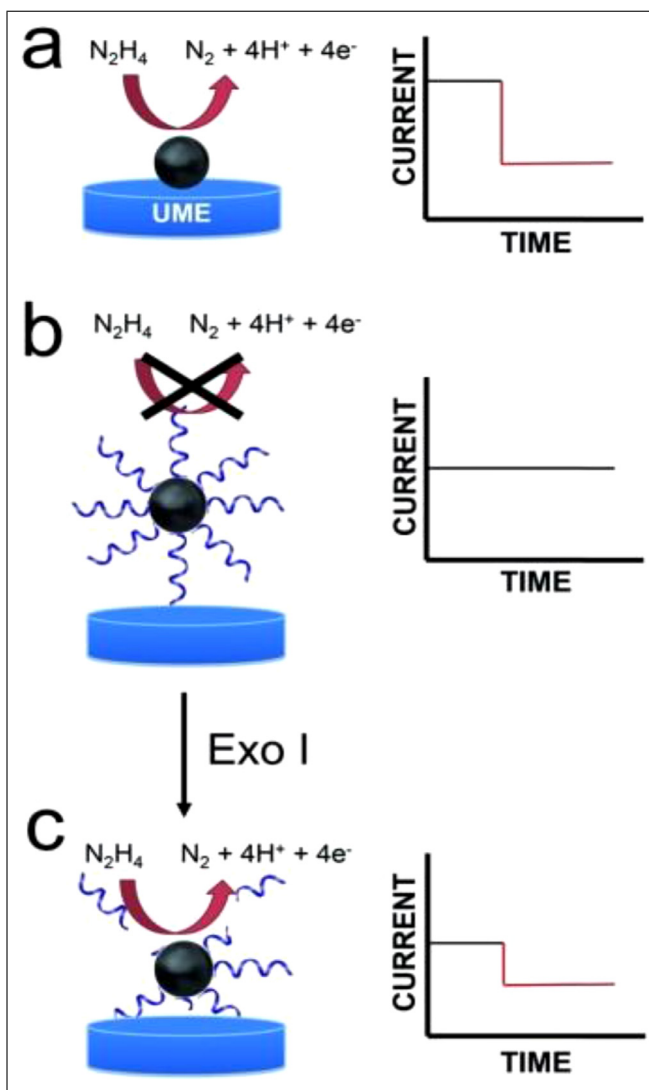
in close proximity to the electrode, the hydrogen peroxide was decomposed back to oxygen by the SiNP/catalase hybrid. As oxygen production of a single hybrid is directly proportional to the amplitude of the peak, the coverage of hybrids over the electrode could be calculated. However due to irreversible adsorption of the hybrids to the glass around the microelectrode used, the frequency of current spikes was less than theoretically calculated [19].

Nanoimpacts have been reported to be able to explore single enzyme catalytic activity, which has the potential to better understand enzyme mechanisms [20,21]. Shleev *et al.* [22] used nanoimpacts resulting from reduction of oxygen by the enzyme laccase, chosen for its relatively well understood structure and because it allows direct electron transfer between its active site and a gold ultra-microelectrode. This direct electron transfer can be measured via nanoimpacts to monitor the current produced from a single enzyme redox reaction, the current produced is amplified because of the enzymes high turnover rate ( $k_{\text{cat}}$ ) [23]. Han *et al.* [20] used modified nanoelectrodes to achieve a staircase current response from the reduction of hydroperoxide with horseradish peroxidase.

### Novel applications

Prussian blue, known for its stable and reversible  $\text{K}^+$ -insertion electrochemistry, was studied at both the single

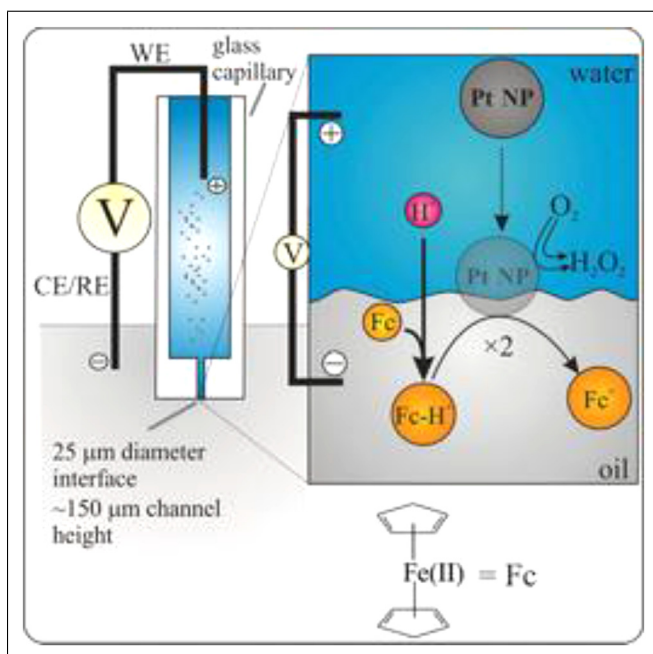
Figure 2



Scheme illustrating how electrocatalytic amplification occurs both with and without the studied enzyme. Adapted from Ref. [18<sup>•</sup>]. Published by the Royal Society of Chemistry.

nanoparticle, and the ensemble level. Impacts were used at the single NP level to allow analysis unaffected by additives and binders [24]. At this level only  $\text{K}^+$  de-insertion occurred which was related to the electrostatic charge between the particle and the electrode surface. These results demonstrated how nanoimpacts can be of use to test the intrinsic electrochemical behaviour without the influence of additives and binders. Single fusion events at polarized liquid–liquid interfaces have also been studied, these events are common in biology [25]. Four different scenarios which can occur upon fusion at an interface between two immiscible electrolyte solutions (ITIES) and nanoimpacts were used to detect an ionic probe which is released upon collision with the interface as well as

Figure 3

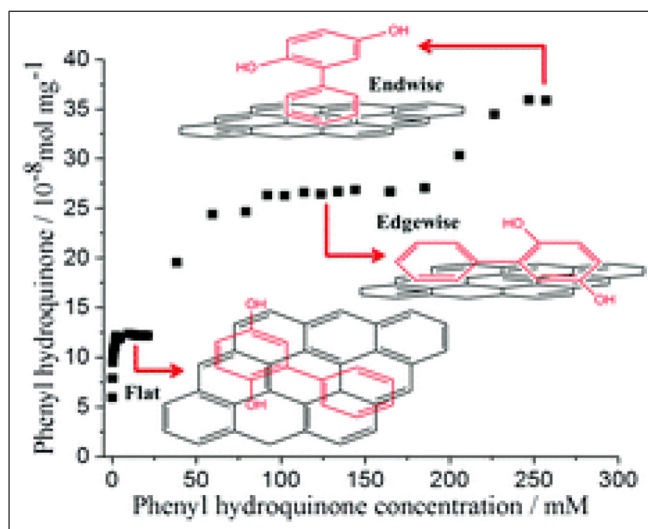


Depiction of Pt NPs collision detected at the ITLES through catalytic ORR [26] © 2017 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

determination of their ionic content and sizing of the particles [25]. The ITIES has also been used to study Pt NP impacts [26]. For the first time metal impacts at a ITIES have been detected by the catalytic ORR of Pt NPs in water when they meet the interface of 1,2-dichloroethane as shown in Figure 3 [26]. The resulting current spikes showed rolling/bounding of the NPs indicating deactivation followed by reactivation without overall catalytic activity loss of the NPs [26]. Other interfaces have also been analysed, such as water droplets in oil [27]. The droplets were tagged with potassium ferrocyanide to allow oxidation of the droplets, peaks created by the oxidation of the ferrocyanide allowed the mean radius of the water droplets to be found [27].

Graphene nanoplatelets (GNPs) have improved stability over graphene, and so were used by Wu *et al.* [28] to be tagged with individual ferrocene derivatives: nanoimpacts were then used to analyse the coverage of the ferrocene on the GNP. Chen *et al.* [29] extended the use of GNP impacts by measuring the kinetics of the adsorption and desorption of catechol from the GNPs. The GNPs were tagged with catechol allowing analysis of its oxidation and the surface coverage to be found. The kinetics of the adsorption were found via electrochemical and spectroscopic methods but the desorption process was assessed by analysing nanoimpacts with two different amounts of catechol at different potentials from 0.2 to 1.2 V. The average charge per spike decreased with time, as this is

Figure 4



PHQ adsorption isotherm for GNPs in solution annotated with the PHQ orientation of each concentration. Reproduced from Ref. [30] with permission from the PCCP Owner Societies.

proportional to the adsorbed catechol, the rate constant was found. Further research investigated the adsorption of phenyl hydroquinone (PHQ) on GNPs finding three different orientations of PHQ [30]. The orientation of the PHQ was found from analysing the peaks to find the surface coverage. With increasing concentration it was found to move from flat to edgewise and then edgewise to endwise orientation, this can be seen in Figure 4 [30] below. At higher concentrations of PHQ the excess modifier required causes too high a background current for spikes to be detected. Adsorption has also been quantified on single insulating particles. Catechol, anthraquinone, p-chloranil and the ferrocene moiety of poly(vinylferrocene) were adsorbed on alumina and the surface coverages and charge diffusion coefficients were found for each. The technique employed both modelling and experimental methods to deduce the monolayer adsorption of these species onto single particles [31].

Nanoparticle porosity has recently been explored using nanoimpacts by tagging Pt NPs on carbon electrodes with 4-nitrobenzenethiol (NTP) [32]. NTP is reduced and the charge of each averaged, to give the number of NTPs, from this the Pt active surface can be found. This was related to two different models, (fully solid and porous NPs) which allows the porosity to be revealed [32]. Jiao *et al.* [33] looked at the direct oxidation of Pt nanoclusters when in contact with the microelectrode, assessing the amount of Pt NPs seeing the electrolyte in the cluster by analysing the nanoimpacts at different potentials and combining these results with XPS. The results showed good correlation and both studies suggest a porous Pt



NP cluster [32,33<sup>\*</sup>]. The current detected on a collector electrode can arise from the surface oxidation of an attached Au NP [34]. The surface oxidation occurs quickly ( $\mu\text{s}$ ) and was used as an alternative method to size NPs and additionally study the surface oxidation of the noble metal, which can greatly affect electrocatalytic activity. Resistance measurements of single carbon nanotube (CNT) electrode contacts have been measured via the nanoimpact of a single CNT bridging the gap between two microbands of a gold electrode. By applying a potential difference between the two microbands, a current is induced across the CNT, this change in current can be measured allowing the resistance of the CNT gold contact to be measured. This work could be useful within fuel cell research as CNTs are often considered for catalyst supports [35]. This was later expanded upon by adding in the electroactive species, acetaminophen [36]. By analysing nanoimpacts with and without the acetaminophen, the group were able to reveal the increase in current amplitude, showing improved contacts between the CNTs and the microbands of the electrode. This suggested improved junction properties, in theory due to redox shuttling [36].

The nanoimpact technique can also be used to investigate electrochemical experiments themselves to ensure optimisation. The use of different capping agents in the preparation of nanoparticles, which is important as drop casting is known to cause agglomeration of the capping agents, change kinetics, and affect the outcome of experiments [37,38]. Tanner *et al.* [37] compared citrate and DNA as capping agents for silver nanoparticles and how this affects the oxidation of Ag at different potentials. DNA capped Ag NPs oxidised at a higher potential than citrate capped Ag NPs. The higher potential corresponds with the oxidation of guanine and adenine in the DNA, suggesting the Ag cannot be oxidised before the DNA. This showed DNA does not allow enough electron transport to allow Ag to be oxidised and so either, once oxidised the DNA starts conducting enough to allow the oxidation of Ag or it is desorbed [37]. Cluster formation can also be an issue in nanoparticle solution due to either aggregation or agglomeration. This is often studied by dynamic light scattering (DLS) though this requires dilution of the nanoparticle solution which can produce distorted results. By using a dye which is reduced when in contact with an electrode to modify rutile ( $\text{TiO}_2$ ) particles and comparing chronoamperometric signals the size of the clusters can be found. The results of this showed much larger groups of clusters than was detected by DLS indicating that nanoimpact measurements are a useful tool for size distribution in nanoparticle solution [39].

## Considerations

Although the technique has proven fruitful in the experiments above, there are still difficulties and factors should be taken into account when considering the method

for applications. Foremost is that background currents must be low and electronic bandwidth electronics high enough to gain a valuable signal to noise ratio. However electrolyte composition and concentration can influence nanoimpact experiments: for example Krause *et al.* [40] found that the response at some potentials can be dependent on the electrolyte conductivity and in the case of Ag nanoparticles on the concentration of chloride ions. It was also shown that the chloride ion concentration drastically affects the current spike shape, which can be explained by the diffusive mass transport of  $\text{Cl}^-$  to the nanoparticle which then limits the oxidation rate of the silver nanoparticles. Colloidal stability within the electrolyte can affect the NP collisions, this has been investigated using Hg UME with Pt NPs [41]. It has shown that aggregation of NPs is increased with the ionic strength of the phosphate buffered hydrazine electrolyte. By ensuring a lower ionic strength of the electrolyte the aggregation of the particles was less of an issue. Highlighting the importance of ensuring the NPs will not be affected by the electrolyte before beginning collision experiments [41]. It was further shown that there is the potential for gases which may be evolved from hydrazine that could destabilise the citrate layer of the Pt NPs allowing aggregation of the NPs [42]. Hg UMEs can be used to reduce the issue in some experiments of signal to noise ratio [43]. Nanoparticle impacts have also now been reported in non-aqueous media, an advantage of this is the lower viscosity of organic liquids such as toluene and dichloromethane which reduces the time of first arrival allowing faster detection [44]. This paper successfully showed Au impacts on a Pt microelectrode in non-aqueous media, these impacts indicated rapid, reversible agglomeration which caused repeated collisions. Another consideration is that particles can get stuck to the glass around the UME which can lead to lower signals than expected [19]. A helpful guide to nanoimpact experiments has been produced which highlights considerations to be made before beginning and what to expect from results [45<sup>\*\*</sup>].

## Conclusions

The work in this paper highlights the advancements in nanoimpact techniques in recent years providing information of nanoscale processes in a diverse range of materials. Though the area still has space for advancement, for example with improvements in DNA analysis, nanoimpacts is a promising technique and research into new applications involving single particles is expected as well as further insights into the structure reactivity relationship.

## Acknowledgement

The authors thank the EPSRC for funding (grant no. EP/G037116/1).

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  - This paper is of outstanding interest as it details how to run an impact experiment.